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DATA EVALUATION RECORD

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Metolachlor

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STUDY ID 41309803

Merricks, D.L. 1989f. Metolachlor field dissipation study in Iowa-Site A. Agrisearch Project No. 1265-A. Unpublished study performed by Agrisearch Incorporated, Frederick, MD, and submitted by Ciba-Geigy Corporation, Greensboro, NC.

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DIRECT REVIEW TIME - 6

REVIEWED BY: L. Binari

TITLE: Staff Scientist

EDITED BY: S. Syslo
K. PattenTITLE: Staff Scientist
Task Leader

APPROVED BY: W. Spangler

TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD
TEL: 468-2500

APPROVED BY: G. Tompkins

TITLE: Chemist

ORG: EFGWB/EFED/OPP

TEL: 557-7346

SIGNATURE: *George Tompkins*CONCLUSIONS:Field Dissipation - Terrestrial

1. This study is considered supplemental at present.
2. This study will be reevaluated upon receipt of an explanation of the following:

The results from the laboratory fortified control samples, fortified at 0.05 to 5.0 ppm (all degradates) or to 10.0 ppm (metolachlor), carried through the procedure with the test samples indicate a wide variation in the recovery efficiencies. These recovery efficiencies ranged from 51 to 202% of the applied for metolachlor, 34 to 137% for CGA-51202, 41 to 166% for CGA-40172, 50 to 158% for CGA-40919, and 15 to 290% for CGA-50720 (Table 7). Also two soil samples from each sampling depth that were spiked in the field at 1.0 ppm showed recoveries ranging from 18.8 to 114.5% (Table 9).

The degradates CGA-40172 and CGA-40919 were included in the field dissipation study. In the aerobic and anaerobic metabolism study (MRID # 41309801-B) under aerobic conditions these two degradates were only detected but not quantifiable. However, other degradates (CGA-41638 was 2.06% of applied at 90 days, CGA-37735 was 1.27% at 30 days, and CGA-13656 was 1.02% immediately posttreatment) were not also included or used in place of degradates that occurred at such a minimal level that they could only be detected but not quantitated (See Comment # 3).

3. Metolachlor dissipated with a registrant calculated half-life of 159 days from the upper 6 inches of silt loam bareground field plot treated with metolachlor (25% G) at 6 lb ai/A. In an adjacent plot treated with metolachlor at 4.0 lb ai/A and then immediately planted to corn, metolachlor dissipated with a registrant calculated half-life of 141 days from the 0-to 6-inch soil layer.

METHODOLOGY:

Metolachlor (Dual 25G, 28% G, Ciba-Geigy) was surface-applied by hand, using a broadcast spreader, at 4 and 6 lb ai/A to two field plots (50 x 50 feet) of silt loam soil (0- to 6-inch layer; 36% sand, 52% silt, 12% clay, 4.6% organic matter, pH 5.8, CEC 15.9 meq/100 g) located in Des Moines, Iowa, on June 4, 1987. One week prior to application, the field had been disked to a depth of 12 inches. The day before treatment, the plots were disked twice to a 6-inch depth; immediately following treatment, the test substance was incorporated by diskings. The plot treated at 4 lb ai/A was planted to corn immediately posttreatment; the plot treated at 6 lb ai/A was left bare. Untreated bareground and corn plots (sizes unspecified) located 150 feet upslope were maintained as controls (Figure 3). Twenty soil cores (5-foot length, 1-inch diameter, stainless steel probe with an acetate liner) were collected prior to treatment; four cores per subplot from three randomly selected subplots were taken at each sampling interval between 0 and 546 days posttreatment. Cores were stored frozen for up to 631 days prior to extraction (Appendix D).

Frozen soil cores were divided into 6-inch increments, and the increments from the four cores per subplot were composited and homogenized. A portion of each composited sample (50 g) was refluxed with methanol:water (1:1) for 1 hour. An aliquot (60 mL) of the extract was mixed with a water:saturated sodium chloride solution, the pH was adjusted to 1-1.5 with 1 N sulfuric acid, and the solution was then partitioned three times with hexane:ethyl acetate (1:1, v:v). The organic phases were combined and dried with anhydrous sodium sulfate; the solution was then methylated with diazomethane. After 30 minutes, the solution was evaporated to dryness, redissolved in hexane, and analyzed for metolachlor and its degradates CGA-51202, CGA-40172, CGA-40919, and CGA-50720 by GC using an OV-17 capillary column and a flame-thermionic detector. The detection limits were 0.05 ppm for metolachlor, CGA-51202, and CGA-40172; 0.06 ppm for CGA-40919; and 0.07 ppm for CGA-50720. Recovery efficiencies from soil samples fortified at 0.05 to 5.0 ppm (all degradates) or to 10.0 ppm (metolachlor) ranged from 51 to 202% of the applied for metolachlor, 34 to 137% for CGA-51202, 41 to 166% for CGA-40172, 50 to 158% for CGA-40919, and 15 to 290% for CGA-50720 (Table 7); the concentrations of metolachlor and its degradates detected in the field soil samples were corrected for recoveries that were <100%.

Selected soil samples were also analyzed for metolachlor and its degradates by GC/MS using single-ion monitoring; the detection limit was 0.1 ng/ μ L.

DATA SUMMARY:

Metolachlor dissipated with a registrant-calculated half-life of 159 days ($R^2 = 0.668$) from the upper 6 inches of a bareground field plot (50 x 50 feet) of silt loam soil in Des Moines, Iowa, that was treated with metolachlor (25% G), at 6 lb ai/A, on June 4, 1987. In the 0- to 6-inch soil depth, metolachlor was detected at an average of 1.83 ppm (maximum 5.93 ppm) immediately posttreatment, 1.95 ppm at 3 days, 0.1 ppm at 22 days, 1.36 ppm at 28 days, and 0.11 ppm at 546 days (final sampling interval) (Table 2).

In an adjacent plot that was treated with metolachlor at 4.0 lb ai/A and then immediately planted to corn, metolachlor dissipated with a registrant-calculated half-life of 141 days ($R^2 = 0.808$) from the 0- to 6-inch soil layer. Dissipation of metolachlor from the upper 6 inches of the corn plot was variable, with an average of 3.01 ppm (maximum 5.70 ppm) immediately posttreatment, 0.37 ppm at 7 days, 1.12 ppm at 28 days, and 0.1 ppm at 546 days (Table 1).

The concentrations of metolachlor degradates detected in the soil were similar for the bareground and corn plots. In the 0- to 6-inch soil depth, the degradate

CGA-51202

was a maximum of 1.05 ppm at 28 days posttreatment;

CGA-40172

was maximum of 0.39 ppm at 91 days; and

CGA-40919

was a maximum of 0.42 ppm at 60 and 91 days (Tables 11 and 12).

Downward movement of metolachlor resulted in a maximum concentration of 1.12 ppm in the 6- to 12-inch soil layer (7 days posttreatment); ≤ 0.06 ppm in the 12- to 18- and 18- to 24-inch depths, 0.12 ppm in the 24- to 30-inch depth, < 0.05 ppm in the 30- to 36-inch depth, and 0.24 ppm in the 36- to 48-inch depth (Table 11). Maximum concentrations of metolachlor degradates in the soil layers below 6 inches were as follows: CGA-51202 was 0.48 ppm in the 6- to 12-inch depth and ≤ 0.07 ppm below 12 inches (up to 48 inches); CGA-40172 was 0.15 ppm in the 6- to 12-inch depth, 0.13 ppm in the 12- to 18-inch depth, 0.93 ppm in the 18- to 24-inch depth, and was ≤ 0.11 ppm below 24 inches; CGA-40919 was 0.31 ppm in the 6- to 12-inch depth, 0.15 ppm in the 12- to 18-inch and 18- to 24-inch depths, and ≤ 0.07 ppm below 24 inches. The degradate CGA-50720 was not detected (< 0.07 ppm) in any soil sample at any interval.

During the study, rainfall totaled 49.9 inches, air temperatures ranged from -19 to 102° F, and soil temperatures (0- to 8-inch depth) ranged from 3 to 114° F.

COMMENTS:

1. It appears that the analytical method was unreliable, especially when approaching the limits of detection ; recovery from fortified samples was unusually low and variable. Recovery efficiencies from spiked samples for metolachlor at 0.05 ppm ranged from 24 to 211% of the applied, and at 0.1-10.0 ppm ranged from 61-159%; for CGA-51202 at 0.05 ppm, recovery efficiencies ranged from 45 to 142%, and at 0.1-5.0 ppm ranged from 33 to 138%; for CGA-40172 at 0.05 ppm, recovery efficiencies ranged from 16 to 179%, and at 0.1-5.0 ppm ranged from 40 to 158%; for CGA-40919 at 0.05 ppm, recovery efficiencies ranged from 36 to 168%, and at 0.1-5.0 ppm ranged from 50 to 139%; for CGA-50720 at 0.05 ppm, recovery efficiencies ranged from 50 to 164%, and at 0.1-5.0 ppm ranged from 27 to 133% (Table 7).
2. The data were variable such that it is difficult to accurately determine the dissipation of metolachlor and its degradates in the soil. In addition to the variability of average concentrations of metolachlor in the soil between sampling intervals, metolachlor residues were also highly variable from sample to sample at the same interval. For example, in samples from the 0- to 6-inch soil layer taken at day 0, metolachlor ranged from <0.05 to 5.7 ppm in the crop plot (Table 11) and from <0.05 to 5.93 ppm in the bareground plot (Table 12).
3. In the aerobic and anaerobic metabolism study (MRID No. 41309801-B) the major degradates of metolachlor under aerobic conditions were: CGA-51202, reaching a maximum of 28.09% of the applied at 90 days posttreatment;
CGA-50720, at a maximum of 14.85% of applied at 272 days;
CGA-41638, at a maximum of 2.06% at 90 days;
CGA-37735, at a maximum of 1.27% at 30 days;
CGA-13656, at a maximum of 1.02% immediately posttreatment. Other degradates that were detected but not quantifiable were CGA-40172, CGA-41507, CGA-40919, and CGA-37913.

In the anaerobic metabolism portion of the same study the major degradate in the soil and flood water was CGA-51202 at a maximum of 23.33% of the applied at 29 days after anaerobic conditions were established. Other degradates isolated from the soil and water were: CGA-41638, at a maximum of 8.30% of the applied at 60 days;
CGA-50720, at a maximum of 7.34% at 60 days;
CGA-13656, at a maximum of 1.46% at 29 days; and
CGA-37735, at a maximum of 1.25% at 29 days.

However, in this field dissipation study metolachlor and its major degradates CGA-51202 and CGA-50720 were analyzed for as well as the degradates CGA-40172 and CGA-40919, which were only detectable but not quantifiable in the above referenced aerobic metabolism study. No explanation was provided by the registrant as to why the other major degradates listed above that were isolated in the aerobic metabolism study were not also included as standards to determine their environmental fate in the field dissipation study.

4. Field soil samples were stored frozen for up to 631 days prior to extraction; however, the stability of metolachlor and its degradates in the soil samples could not be confirmed because the available storage stability data were too variable. In a storage stability experiment conducted with soil taken from the control plot at the test site, soil samples (6-inch increments from depths of 6 inches to 60 inches) were fortified with metolachlor and the degradates CGA-51202, CGA-40172, and CGA-40919 at 1.0 and 5.0 ppm, then stored frozen (temperature not specified) for up to 901 days. After 70 days of storage, recoveries ranged from 8.7 to 125.9% of the applied; after 538/539 days, recoveries ranged from 11.0 to 133.3%; and after

901 days, recoveries ranged from 44.9 to 149.7% (Table 8). In addition, results from reanalysis of field soil samples could not consistently be replicated; for example, an average concentration of 1.64 ppm of metolachlor (range 1.44-1.94 ppm) was detected in 3-day soil samples (0- to 6-inch depth) from the crop plot that were extracted 8/17/87, but when the soil samples were reanalyzed 2/8/89, an average concentration of 0.73 ppm of metolachlor (range <0.05-1.50 ppm) was found (Table 11; Appendix D). The wide variability in the storage stability data and the results from the reanalysis of field samples may have been due to the inability of the method to accurately determine metolachlor residues.

In addition, the storage stability of the degradate CGA-50720 in soil samples fortified at 1.0 ppm was investigated at only one interval; after 127 days of storage, 51.1-107.5% of the applied was recovered. The 127-day sampling interval is inadequate since the analytical method was not modified to isolate CGA-50720 until the 317-day field soil samples were analyzed (see Comment 5).

5. Prior to analysis of the 317-day soil samples, the analytical method was modified to recover a fourth degradate (CGA-50720). The method description for the soil extraction was not included in the report proper, but was included in the Protocol (Appendix A). The method summarized in this report is the final modified method.

In addition, although not specifically stated, it appears from the extraction dates (Table 7) that all relevant soil samples were reanalyzed using the modified method.

6. Air and soil temperature data were obtained from the NOAA weather station at Ames, Iowa; the distance from the weather station to the test site was not reported. Rainfall data were collected at the test site from June 4 to September 30, 1987, then for the remainder of the study, the data were obtained from the weather station. EPA prefers that meteorological data be taken at the test site.
7. The depth to the water table was 5-8 feet, and the slope of the field was <1 degree. Field maintenance practices of the treated plots during the study were not reported.
8. Analysis of pretreatment soil samples was not provided; however, data from control plot soil samples (Table 10) indicate that metolachlor was not present prior to study initiation.
9. The plots had been planted to oats prior to the study; no chemicals were applied "for more that 12 months prior" to the study.
10. Two soil samples from each sampling depth were spiked in the field at 1.0 ppm; samples were sealed, frozen on dry ice, and stored frozen (temperature not specified) for 56 days before analysis. Recoveries ranged from 18.8 to 114.5% (Table 9).

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Pages 6 through 71 are not included.

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- ☐ Identity of product inert ingredients.
- ☐ Identity of product impurities.
- ☐ Description of the product manufacturing process.
- ☐ Description of quality control procedures.
- ☐ Identity of the source of product ingredients.
- ☐ Sales or other commercial/financial information.
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